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DOCTORAT THESIS ABSTRACT

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SUMMARY

STUDIES AND EXPERIMENTAL RESEARCHES ON THE DEFORMABILITY AND BEHAVIOR OF CAVITATIONAL EROSION OF INCONEL-718

THANKS

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INTRODUCTION

The development of humanity has always been based on the great achievements of materials and their performance, each stage of which is marked by a leap made in the field of materials, in general, of metallic materials, in particular. The wide spectrum of properties and uses of metallic materials has determined a permanent development of experimental research for the discovery of new performances, new applications of metallic materials. Within metallic materials, superalloys are a permanent attraction even today due, on the one hand, to the identification of new performances, and, on the other hand, to the development of new methods of their investigation.

Cavitative erosion is a harmful effect on cavitation, regardless of where it acts. Since it manifests itself differently from one material to another, depending on the resistance it opposes to cavitational attacks, one of the concerns of scientists is to establish the causes that determine this resistance.

Looking from the perspective of these aspects, respectively superalloys and possibilities of exploring their properties, this doctoral thesis is part of the current concerns. The general objectives of the doctoral thesis consist in the following aspects:

- Determining the behavior of hot plastic deformation, in dynamic laboratory conditions of the INCONEL 718 superalloy in order to identify its mechanisms, the structural characterization of the material and to determine the optimal processing parameters of the superalloy.
- Determining the behavior of cavitation erosion of the INCONEL 718 superalloy, in laboratory conditions, both in delivery and in different states of sensitization in order to identify the mechanisms of propagation of the cavitational phenomenon, as well as the comparative highlighting of the superalloy resistance state INCONEL 718 with the resistance state of austenitic stainless steels, whose behavior is well defined in current research.

In order to meet the proposed objectives, structural investigations were performed on INCONEL 718 specimens, taken over from Carpenter Technology Corporation. In order to determine the deformability behavior, the experimental researches were performed on a free-falling hammer (sonnet) from the "Forging, foundry and extrusion" laboratory of the Faculty of Materials Science and Engineering at the Polytechnic University of Bucharest. The height of fall used was H = 0.5 m and mass m = 71 kg; using three samples for each test temperature in the range 800-112°C, the determinations being made every 50°C. The heating of the samples for plastic deformation was carried out in an oven with forced heating rods, located in the immediate vicinity of the test apparatus. The samples from the same casting batch were cut by EDM, having the initial diameter d0 =10 mm and the height h0 = 15 mm. The main data that were taken into account in the experimental determinations were: upper hammer weight G = 695.8 N (m = 71 kg); hammer drop height H = 0.5 m; coefficient of friction $\mu = 0.3$ (hot, without lubricant); the yield of the free-falling hammer was determined by the Heim method, it was approximately $\eta = 0.7$ (70%). For these investigations, the specimens were studied quantitatively and qualitatively under a metallographic optical microscope, in different states, X-ray diffraction analysis was performed and the μHV200 microd hardness values were determined. Using the EBDS technique, the structural characteristics of the specimens required for deformability under conditions of free fall of the sonnet were determined

In order to determine the cavitation behavior of the INCONEL 718 superalloy, tests were performed in the cavitation laboratory within the Politehnica University of Timișoara. The test and processing procedure of the results was in strict accordance with the provisions of ASTM G32-2010, respecting the laboratory custom of the total test duration of 165 minutes, divided into 12 intermediate periods (one of 5 and 10 minutes and 10 of each 15 minutes each). The evaluation of the behavior and resistance to erosion by vibrating cavitation was performed by plotting the curves of the cumulative mean depths MDE (t) and the curves of the average penetration rates of erosion MDER (t), and subsequently the specimens required for cavitation were evaluated structurally, quantitatively and qualitatively. by stereomicrostructural analysis, analysis by metallographic optical microscope and analysis by scanning electron microscope. In order to determine the intensity of the cavitation erosion phenomenon of the INCONEL 718 superalloy, comparative tests were performed on an austenitic stainless steel type X5CrNi18-10, in different states of sensitization, as well as the experimental superalloy, all being compared with the resistance to cavitational erosion of OH12NDL standard steel, stainless steel used in the manufacture of Kaplan turbine blades at the Iron Gates Hydropower Plant, appreciated as having good resistance to erosion caused by the vibrating cavity generated by the piezoceramic crystal device.

The thesis includes 5 chapters, as follows:

Chapter 1 contains general information on the development of nickel-based superalloys, in general, the INCONEL 718 superalloy, in particular, documentary research on plastic deformation behavior investigated worldwide and documentary research on the behavior of cavitation erosion of various nickel superalloys.

Chapter 2 presents the research material and methodology, being presented at the end of the program, his own experimental research conducted by the author.

Chapter 3 includes the structural analysis of the INCONEL 718 specimens used in our own experiments. During the experiments were analyzed structurally both the specimens that were required for hot plastic deformation by free fall of the sonnet from different heights, at different temperatures, and the specimens that were required for cavitation, in different structural states of sensitization. Within these structural analyzes, X-ray diffraction analyzes, analyzes under a metallographic optical microscope, as well as the determination of microdurity values at a microdurimeter were performed. Also, grain size measurements were performed, making different structural correlations.

Chapter 4 presents the experimental research on determining the plastic deformation behavior of the experimental superalloy INCONEL 718. After performing laboratory tests, multiple structural comparative analyzes were performed to formulate recommendations on the optimal parameters for the plastic deformation of this superalloy. Thus, the breaking surfaces were investigated under a stereomicroscope, the curves regarding the resistance to plastic deformation depending on the heating temperature, the specific mechanical work depending on the superalloy heating temperature and the variation of the permissible degree of deformation depending on the heating temperature were constructed. An extensive analysis was performed on these specimens under a scanning electron microscope using scattered scattering secondary electrons (SEM-EBSD), determining the grain size, phase distribution and preferential crystallographic orientation (texture), determining the disorientation of the grains / sub-grains. At the end of the chapter, structural correlations were made regarding the variation of the phase (γ, γ', γ'' and M_xC_y)

depending on the deformation temperature of the ICONEL 718 specimens, after the deformability tests.

Chapter 5 presents the cavitation behavior of the experimental INCONEL 718 superalloy, compared to that of some austenitic stainless steels. After construction of the curves of variation of the penetration rate of the MDER erosion (t) and of the average erosion depth (in different structural states, respectively: delivery state and sensitization states at 830°C / 5, 10 and 20 hours) depending on duration of exposure, the data were compared with the results of structural analyzes at stereomicroscope, optical microscope and scanning electron microscope. The same investigations were performed on an austenitic stainless steel X5CrNi18-10 and compared with standard steel OH12NDL. At the end of the chapter, an evaluation of the cavitation resistance of the INCONEL 718 superalloy is made, compared to the cavitation resistance of some stainless steels, constructing two comparative histograms, respectively of the "vibration cavitation resistance of the INCONEL718 superalloy in different structural states. OH12NDL standard steel and X5CrNi18-10 austenitic stainless steel, in different sensing states ", as well as the values of the maximum cavitation depths of the INCONEL718 superalloy in different structural states compared to those corresponding to different austenitic stainless steel X5CrNi18-10 states ".

Chapter 6 summarizes the final conclusions, the author's contributions to the subject, as well as the proposal of further research directions. The part of the original contributions begins with the presentation of the research strategy, indicating the proposed objectives.

The original results were partially capitalized by publications in ISI journals, as well as by presentations at national and international conferences.

CHAPTER 1
CURRENT STAGE OF THE DEVELOPMENT OF INCONEL ALLOYS

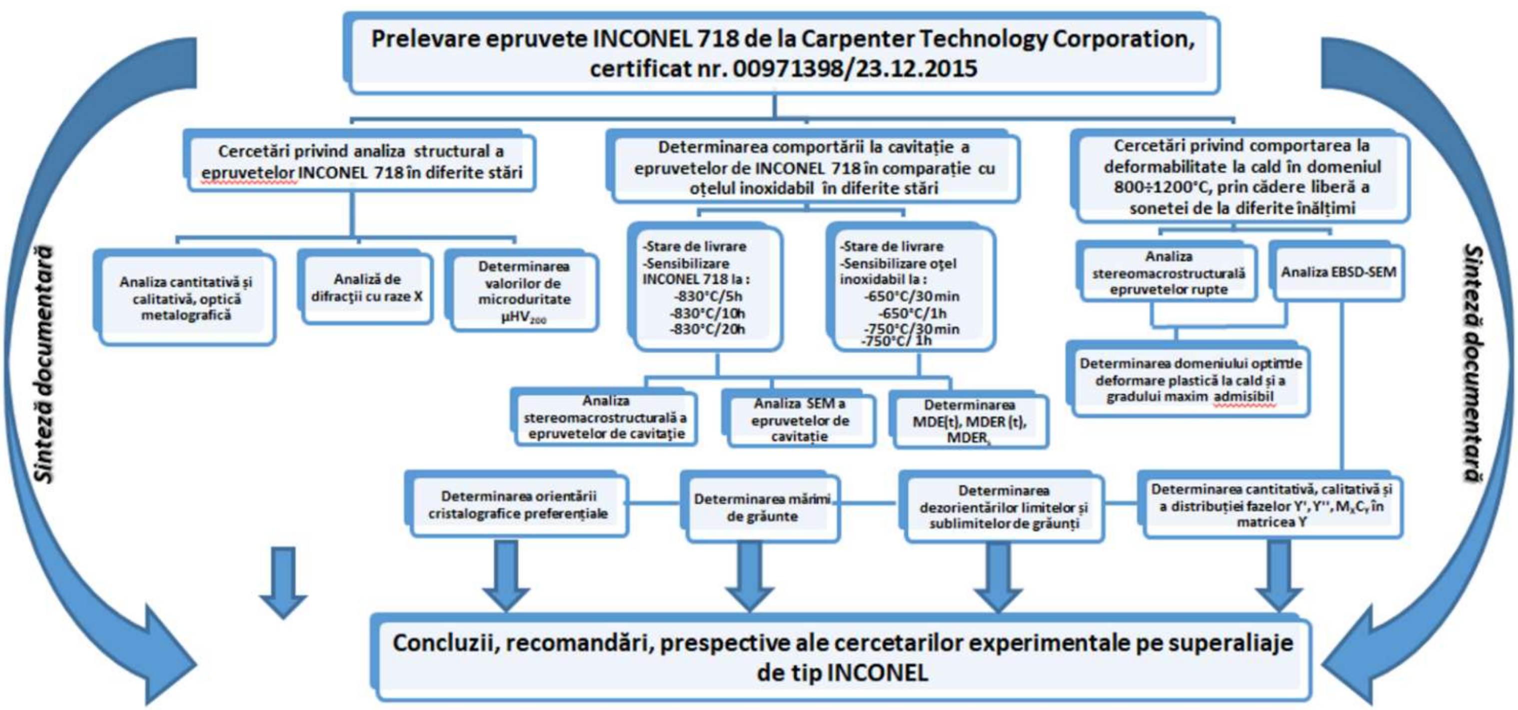
High temperature resistant metallic materials, commonly referred to as superalloys, are developed to meet the challenges of energy efficient requirements, but at the same time minimize greenhouse gas emissions. According to [1, 2], high temperature resistant materials must simultaneously possess the following characteristics;

I. „Ability to withstand a load at operating temperature close to its melting point".
II. „Substantial resistance to mechanical degradation over a long period of time".
III. „Tolerance to severe operating environments".

INCONEL is a registered trademark of Special Metals Corporation for a family of nickel-chromium austenitic superalloys [30]. INCONEL alloys are materials resistant to oxidation and corrosion, suitable for use in extreme environments subject to pressure and heat. When heated, INCONEL forms a thick, stable passive oxide layer that protects the surface from subsequent attacks. INCONEL retains its resistance over a wide range of temperatures, suitable for high temperature applications, where aluminum and steel would fail. The high temperature resistance of INCONEL alloys is developed by solid solution hardening or precipitation hardening, depending on the alloy [31]. INCONEL alloys are commonly used in high temperature applications.

CHAPTER 2
RESEARCH MATERIAL AND METHOD

The research material is produced by Carpenter Technology Corporation on 23.12.2015, with inspection certificate 3.1 EN 10024, certificate number 00971398. The material was developed in induction furnace in vacuum controlled environment, with batch 603205 The experimental program is designed to meet the objectives originally proposed. The two objectives initially proposed, namely the determination of the optimal deformability range of the INCONEL 718 superalloy and the determination of the cavitation behavior of this superalloy were preceded by the structural investigations performed on the INCONEL 718 specimens, taken over from Carpenter Technology Corporation. For these investigations, the specimens were studied quantitatively and qualitatively under a metallographic light microscope, in different states, X-ray diffraction analysis was performed and the microdurinity values determined μHV200.



CHAPTER 3
MICROSTRUCTURAL ANALYSIS OF INCONEL 718 TEST PROBES USED IN OWN EXPERIMENTS

The structural analysis of the investigated materials is of utmost importance in assessing their operating behavior. In the experiments of this paper were structurally analyzed both the specimens that were required for hot plastic deformation by free fall of the sonnet from different heights, at different temperatures, and the specimens that were required for cavitation, in different structural states of awareness. Within these structural analyzes, X-ray diffraction analyzes, analyzes under a metallographic optical microscope, as well as the determination of microdurity values at a microdurimeter were performed.

Microstructural aspects of the specimens required for hot plastic deformation by free fall of the sonnet from different heights, at different temperatures in both sampling sections (longitudinal / transverse).

CHAPTER 4
THE PLASTIC DEFORMATION BEHAVIOR OF EXPERIMENTAL SUPERALLOY INCONEL 718

The plastic deformation behavior performed in laboratory conditions was highlighted by performing experiments on the sonnet from the endowment of the Plastic Deformation Materials Processing laboratory, from the SIM-UPB faculty, and the quantitative and qualitative identification of the phases present in each state was performed. by SEM-EBSD analysis.

Based on the obtained results, graphs of the variation of the resistance to deformation - temperature or specific mechanical work - temperature were made. All data were obtained taking into account the return of the hammer after free fall $\eta = 0.7$ (70%) (Heim method), corresponding to the fall height of approximately H = 0.5 m. It should also be noted that in the case of present, the coefficient of friction of the material at deformation and external working tools was considered as $\mu = 0.30$ (hot plastic deformation without lubrication). The weight of the hammer that fell was G = 695.80 N corresponding to a mass of m = 71 kg.

The experimental results in table 4.2 were processed to represent the graphs of deformation resistance as a function of temperature (figure 4.12) and specific mechanical work as a function of heating temperature for the experimental INCONEL 718 superalloy (figure 4.13).

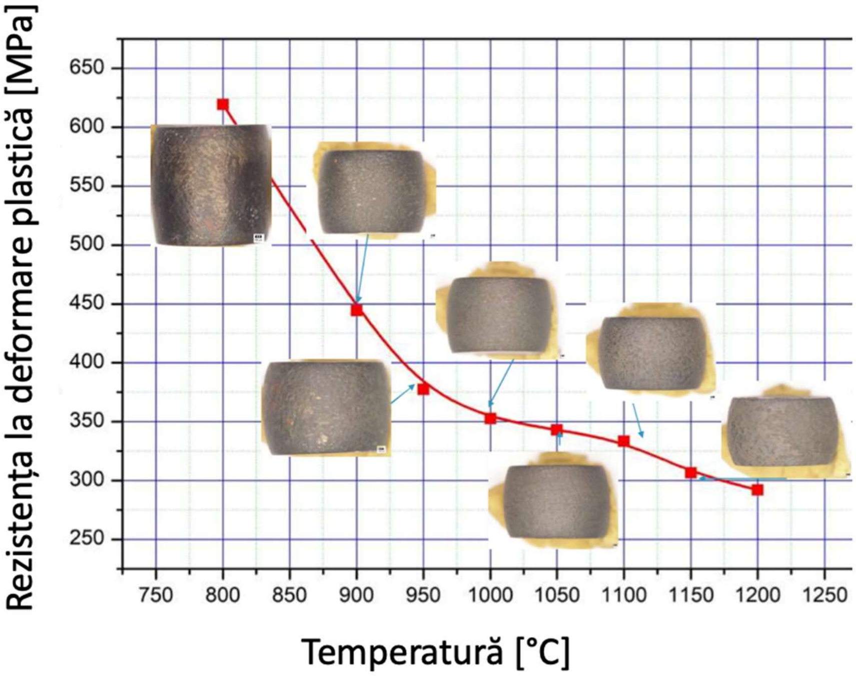


Figure 4.12 - Deformation resistance of plastic as a function of the heating temperature of the experimental superalloy INCONEL 718 [151]

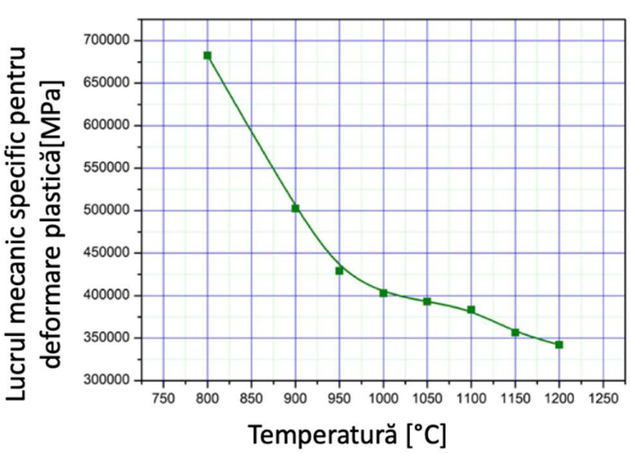


Figure 4.13 - Specific mechanical work as a function of the heating temperature of the INCONEL 718 experimental superalloy after deformability tests [151]

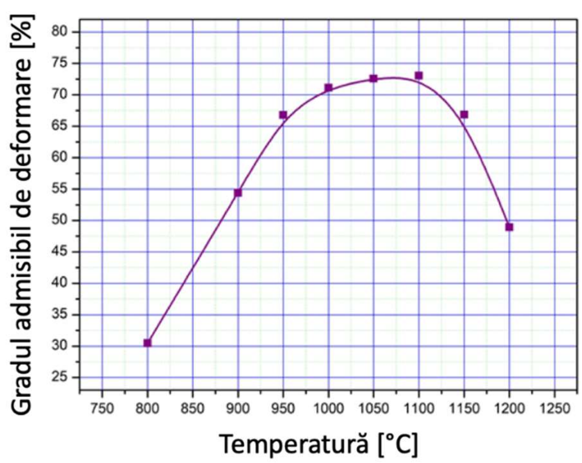


Figure 4.14 - Permissible variation of the degree of deformation as a function of the heating temperature of the INCONEL 718 experimental superalloy after the deformability tests [152]

CHAPTER 5
CAVITY BEHAVIOR COMPARISON OF EXPERIMENTAL INCONEL 718 SUPERALLOY AND OTHER AUSTENITIC STAINLESS STEELS

The histograms below show the values obtained from experimental tests for the average erosion depth and the average rate of erosion penetration, as well as the approximation / mediation curves of these values MDE(t), respectively MDER (t), which characterize the behavior the surface layer at the attack of the cavity.

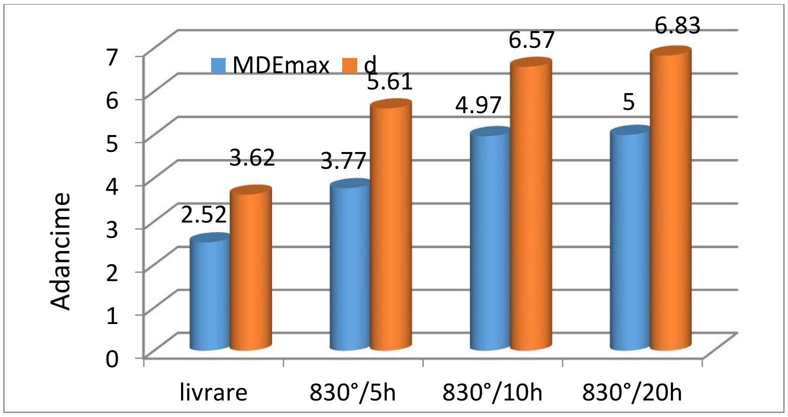


Fig. 5.15. Histogram of depth values of cavitational erosion in INCONEL 718 specimens, in different structural states

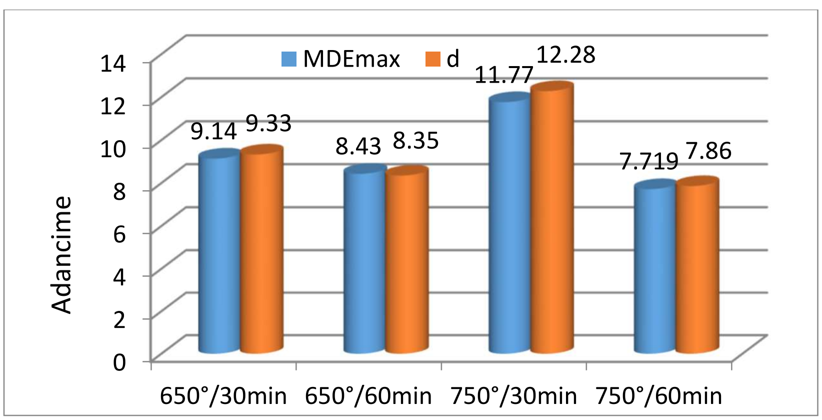


Fig. 5.38- Histogram of cavity erosion depth values for X5CrNi18-10 austenitic stainless steel specimens, in different structural states

CHAPTER 6
CONCLUSIONS, ORIGINALCONTRIBUTIONS, DIRECTIONS AND PRESPECTIVES OF FUTURE RESEARCH

6.1 CONCLUSIONS

The researches carried out within the present doctoral thesis led to the obtaining of some results that allowed to reach the initially proposed objectives, respectively: Determination of the hot plastic deformation behavior of the INCONEL 718 superalloy under dynamic laboratory conditions in order to identify its mechanisms, the structural characterization of the material and to determine the optimal processing parameters of the superalloy, - Determining the behavior of cavitation erosion of the INCONEL 718 superalloy, in laboratory conditions, both in delivery and in different states of sensitization in order to identify the mechanisms of propagation of the cavitation phenomenon, as well as the comparative highlighting of the resistance state of the INCONEL superalloy 718 with the resistance state of austenitic stainless steels, whose behavior is well defined in current research.

6.1.1 Final conclusions from research on structural investigations

The structural analysis of the investigated materials is of utmost importance in assessing their operating behavior. In the experiments of this paper were structurally analyzed both the specimens that were required for hot plastic deformation by free fall of the sonnet from different heights, at different temperatures, and the specimens that were required for cavitation, in different structural states of awareness. Within these structural analyzes, analyzes were performed under a metallographic optical microscope, X-ray diffraction analyzes, as well as the determination of microdurity values at a microdurimeter.

- The microstructural aspects of the specimens required for hot plastic deformation by free fall of the sonnet from different heights, at different temperatures highlighted the identification of specific phases of the material and the evolution of their proportion with temperature and severe plastic deformation conditions. Thus, at a temperature of 800°C, the material has an austenitic structure, with relatively uniform grains, twinned, differently colored, due to their different crystallographic orientation, with a wavy appearance of the image, due to a state of strong deformation of the material. At 950°C the presence of the phenomenon of dynamic recrystallization is noticed, with very fine grains, with crystallographic orientation in the direction of severe compression of the material (in longitudinal section) and with a less oriented orientation (in cross section). At high magnification, NbC particles are highlighted, with intragranular precipitation in the matrix of the metallic material. At a temperature of 1000°C, the dynamic recrystallization is almost in the whole material, with the orientation in the direction of compression of the new recrystallized grains (longitudinal section) and without crystallographic orientation of the grains (in cross section). The NbC and γ'' particles are finely dispersed in the deformed and recrystallized matrix of the material. At a temperature of 1100°C, the recrystallization phenomenon is also maintained throughout the material, without cirstalographic orientation of the grains (either in longitudinal section or in cross section). At 1200°C the phenomenon of grain growth begins, together with the recrystallized, fine grains. Thus, the large grains are oriented in the direction of severe compression, this time in both sections and sampling. The same intragranular precipitates are highlighted at high magnification powers.

- The microstructural analysis of the INCONEL 718 specimens in different sensitization states at 830°C, with maintenance times of 5 hours / 10 hours / 20 hours compared to the appearance of the specimens in the delivery state did not show visible changes under the microscope. All specimens have the specific appearance of austenite twinned grains, uniform, differently colored, depending on their different crystallographic orientations. At high magnification powers are highlighted MxCy carbides (either fine, globular black or orange, geometric NbC), with intragranular precipitation.
- X-ray diffraction analysis. With the appearance of diffractograms at different temperatures should have highlighted in addition to the austenitic matrix (γ, Ni-Cr-Fe) whose maximum intensities are specified at the corresponding diffraction angles and phases □□ , □□□, MxCy. This could not be achieved due, on the one hand, to the fact that the proportion of each phase is very small, their sum being less than 5 ÷ 10%, or, on the other hand, to the Cu and not Mo anticathode tube, which probably would have allowed the detection of these phases.
- Determination of microd hardness values μHV_{200} allowed the observation of a correlation between the temperature at which the hot plastic deformation is made. Thus, it is noted that by increasing the temperature of hot plastic deformation in the range 900° ÷ 1200°C the micro hardness μHV_{200} decreases, the possible mechanisms of influence being on the one hand the recrystallization phenomenon, with maximum intensity at 1050°C (so minimum micro hardness) and, on the other hand, the precipitation of the γ′′ phase, which facilitates hot plastic deformation. The application of different heat sensitization treatments at 830°C of INCONEL 718 specimens showed that increasing the sensibilization time from 5, 10 hours and 20 hours can reduce by about 20% the values of microhardness.

6.1.2 Final conclusions from the research on determining the deformability of the INCONEL 718 superalloy

The plastic deformation behavior performed in laboratory conditions was highlighted by performing experiments on the sonnet from the endowment of the Plastic Deformation Materials Processing laboratory, from the SIM-UPB faculty, and the quantitative and qualitative identification of the phases present in each state was performed. by SEM-EBSD analysis. After conducting preliminary research on the free fall distances as a function of the test temperature, the specimens without cracks were selected, at which the values of the deformation resistances were calculated, as well as the values of the maximum permissible degrees of hot plastic deformation.

- The macrostructural analysis of the breaking surfaces of the INCONEL 718 specimens subjected to plastic deformation by free fall of the sonnet from different heights, highlighted the following aspects:
  - Propagation of the rupture front occurs in all specimens on a plane inclined at 45°, in accordance with Schmid's law,
  - The aspects of the breaking surfaces in a section perpendicular to the direction of compression show a ductile behavior, with gaps evenly distributed in the surface, with a specific coloration depending on the crystallographic direction of the blue-violet breaking planes (in specimens at 950°C and, the temperature 1000°C). The rupture has a ductile character, with transgranular propagation.
  - The appearance of the specimens after heating and testing at the sonnet is oxidized, compared to the shiny appearance of the initial specimens. There is also a brown color with different colors (due to the specific alloying of the alloy)
- After analyzing the variation graphs of the two parameters $\Delta\sigma'_c$ and A with respect to the heating temperature, it can be observed that with the increase of the temperature, the resistance to plastic deformation of the INCONEL 718 superalloy decreases in the investigated temperature range. Similarly, the specific mechanical work done on plastic deformation decreases considerably with increasing temperature. In other words, the data obtained showed that both the yield strength and the mechanical work specific to deformation decrease with increasing temperature, as a curve whose slope varies with the experimental temperature. Both the deformation resistance and the specific mechanical work of hot deformation decrease approximately to 950°C, have a bearing appearance in the range of 950°C ÷ 1050°C, than to begin to decrease slowly; this behavior is probably due to intergranular compounds that begin to melt at low temperatures. Compared to other alloys or classes of superalloys or from other systems, it can be considered that the Inconel 718 superalloy has a rather high localized deformability range, having a ductile breaking behavior and a bright transcrystalline appearance. The following can be concluded as follows:
  - The optimal temperature range for hot plastic deformation of the Inconel 718 superalloy, strictly in terms of deformation resistance, is in the range of 950° ÷ 1050°C, because the values of deformation resistance of the material and the specific mechanical work of deformation are optimal;
  - Both the resistance to deformation and the specific mechanical work of hot deformation decrease approximately to 950°C, have a linear appearance in the range of 950°C ÷ 1050°C, and begin to decrease slowly; this behavior is probably due to intergranular compounds that begin to melt at low temperatures;
  - The degree of formation allowed is very high in the range of 1050° ÷ 1100°C, around 70% -75%, with a steep increase to a temperature of 950°C;

- Compared to other alloys or classes of superalloys or from other systems, the Inconel 718 superalloy can be considered to have a fairly high range of localized deformability, with a ductile breaking behavior and a bright transcrystalline appearance.

➢ Analysis by scanning electron microscope using backscattered secondary electrons (SEM-EBSD) of INCONEL 718 specimens required for compression by deformability tests in laboratory conditions led to results that confirm the stereomacrostructural observations, to determine the values of phase distributions identified, respectively γ, γ′, γ″ and MxCy, as well as when determining the average grain size values, for both sampling directions, longitudinal and transverse. Thus the following can be concluded:

- The analysis of the structural aspects related to the grain size showed the way the material behaves in compression conditions at different temperatures. At 800°C, after the free fall of the sonnet from 0.8 m, both in the longitudinal direction and in the transverse direction, the grains are strongly deformed, flattened, widened and elongated, having a rather uneven distribution, with average values of size of grain from 7 ÷ 8μm, up to 60μm. At this temperature, the highest values of the average grain size are observed, respectively 37.6μm / longitudinal section and 52.3μm / cross section. At 900°C, after the free fall of the sonnet from 1.2m, begins the phenomenon of dynamic recrystallization, by forming a new generation of very fine grains (respectively 2 ÷ 5μm in both sections), along with large grains of 30 -50μm) strongly deformed plastically, elongated, or flattened, at which very large grains can be found, over 65μm. At this temperature, values of the average grain size are observed lower than at 800°C, respectively 22.2μm / longitudinal section and 46.7μm / cross section. At a temperature of 950°C, after the free fall of the sonnet from 1.4 m, the phenomenon of dynamic recrystallization becomes dominant, over 80% of the grains being practically recrystallized, which is observed in both sections. At this temperature, values of very small average grain size are observed, lower than at 900°C, respectively 6.58μm / longitudinal section and 8.8μm / cross section. At a temperature of 1000°C, after the free fall of the sonnet from 1.65 m, the phenomenon of dynamic recrystallization takes place in the entire volume of the specimen, which is observed in both sections, observing values of the average size of small grains, respectively 9.85 μm / longitudinal section and 9.35μm / cross section. At a temperature of 1050°C, after the free fall of the sonnet from 1.66m, the whole volume of material shows dynamic recrystallization, which is observed in both sections with values of the average size of very small grains, respectively 8.72μm / longitudinal section and 9.11μm / cross section. At a temperature of 1200°C, after the free fall of the sonnet from 0.55m, the phenomenon of grain growth begins, a phenomenon observed both in longitudinal section (with large elongated grains) and in cross section (with large widened grains). At this temperature, values of the average size of larger grains are observed, respectively 16.86μm in longitudinal section and 17.43μm in cross section. The incidence of large grains increases, observing grains up to 30μm (in longitudinal section) and about 28μm (in transversal section).

- The quantitative evolution of the proportion of phases in the thermomechanically required material in the temperature range 800° ÷ 1200°C showed that the highest amount of ***austenite γ***, respectively 93.1% / longitudinal section and 94% / cross section, while at 900°C the smallest amount of austenite is observed. At the other temperatures, the amount of austenite is between 83.23% ÷ 86%, in the longitudinal section and 85.9 ÷ 88% in the cross section. ***Phase γ′*** (consisting of Ni3Al and Ni3Ti) begins to precipitate in large quantities at 900°C (7.25% / cross section and 8.98% / longitudinal section), and then stabilizes at lower values, between 5.007% ÷ 5.62% / cross section and 5.73% ÷ 6.39 / longitudinal section at temperatures between 950° ÷ 1200°C. At the temperature of 800°C phase γ′ is at the lowest value, respectively 2.402% / cross section and 2.73% / longitudinal section. The quantitative evolution of the ***γ″ phase*** (Ni3Nb) shows a trend similar to that of the γ′ phase. Thus at 800°C in the matrix there are only small quantities, respectively 1.073% / longitudinal section and 1.17% / cross section, so that in the temperature range 900° ÷ 1200°C this phase has an almost double quantity, respectively 3.23% ÷ 4.32% / cross section and 4.46 ÷ 4.48% / longitudinal section. The behavior of MxCy carbides in the studied field shows that they are in small proportion (about 2%), except for the temperature of 900°C, at which the most carbides precipitated at the grain limit are recorded, respectively 12.34% / longitudinal section and 5.4% / cross section.
- The crystallographic orientation of the material during compression, with the help of pole figures, at high temperatures allowed to highlight the behavior of the material. Thus the phenomenon of dynamic recrystallization, which takes place in the range 900° ÷ 1050°C weakens in intensity the texture of the material (maximum values being in the range 10 ÷ 33 / longitudinal section and 5.2 ÷ 34 / cross section), compared to the values of at high temperature, 1200°C (reaching a maximum value of 50 / longitudinal section).
- The preferential orientation (texture) highlighted by the images of poly iverses in the temperature range 900° ÷ 1050°C in which dynamic recrystallization takes place in the material, is reflected by the values of maximum intensities, respectively 3700-8900 / longitudinal section and 2200- 5600 / cross section, intermediate values from 800°C (16000 / longitudinal and 7900 / transversal) and the lowest values from 1200° (respectively 1800 / longitudinal and 2400 / transversal).

- The analysis of the degree of disorientation of the grains / sub-grains in the material required for compression at high temperatures faithfully reflected its nature. Thus, at low temperature (800°C) or at high temperature (1200°C) there are clearly mixed grain / sub-grain structures. At 800°C the mixed character of the degree of disorientation is given by the phenomenon of strong compression outside the optimal range of plastic deformation, there is even a trimodal model, both subgranules at small angles 8-10° and those at intermediate angles, about 30°, coexists with grain boundaries at large angles, about 50°. At 1200°C, however, at a very high temperature at which the phenomenon of grain growth begins after dynamic recrystallization, the 15°-oriented sub-grains coexist almost equally with the 45-50°-oriented grains, with a bimodal distribution character. the degree of disorientation of the grains. At all other temperatures, due to the phenomenon of dynamic recrystallization, the amount of sublimits at small angles decreases due to rearrangement and migration of boundaries.

6.1.3 Final conclusions from the research on the determination of the cavitation behavior of the INCONEL 718 superalloy

The experimental results on the cavitation behavior [determined by constructing the MDE (t), MDER (t) curves] of the INCONEL 718 experimental superalloy in different structural states were presented comparatively, either quantitatively in graphical form, or qualitatively and quantitatively after the fractographic structural analysis at stereomicroscope, optical microscope and scanning electron microscope. The following conclusions were drawn:

- The analysis of the curves MDE (t), MDER (t) showed the following aspects:
  - the MDE (t) approximation curves have exponential variations, with linearization tendencies, starting from different durations, depending on the structural state of the surface and the hardness values. Thus, it was observed the increase of the duration from which the MDE curve (t) becomes linear as the sensitization duration increases, from 10 hours to 20 hours;
  - the MDER curves (t) for approximating the experimental values recorded for the erosion penetration rates, showed identical evolutions, reaching a maximum, MDERmax and asymptotic decrease towards the stabilization value, MDERs. The differences between the maximum and stabilization values are very small. This mode of evolution is specific to materials with high resistance to cavitation erosion, characterized by high plasticity, mechanical properties (especially mechanical strength, yield strength and hardness) with high values, well correlated, which ensures increased resilience to the surface layer, without turn it into a fragile one;
  - on the interval 0-45 minutes were observed large differences of the experimental values, of the MDER speeds, compared to the approximation curves. From the

experience of the Cavitation Laboratory of the Polytechnic University of Timișoara, these large and random dispersions are determined by roughness, abrasive dust left after the surface finishing operation and the expulsion of large parts of grains by joining cracks produced by impact shocks with microjets and waves. shock;

- the maximum values of the speeds obtained experimentally MDERmax / exp are equal to those recorded by the approximation curve MDERmax and, for the samples made from INCONEL 718 heat treated, regardless of the maintenance duration. The cause is given by the connection between the structure and the hardness of the material in the cavity layer;
- the dispersion of the experimental values compared to the MDER approximation curve (t), after 45-60 minutes, is almost symmetrical, specific to the surfaces whose attacked layer hardens during cavitation, under repeated impact with microjets and shock waves generated by the hydrodynamic mechanism of bubble implosion;
- the experimental values, of the MDER parameter, from the first 45 minutes, are caused by the morphological structure of the material in the delivered state, but also by the fact that the hardness is not constant in the area of the exposed surface;
- as the duration of the cavitation attack increases (see images in table 5.1) the destroyed area increases;

➢ Macroscopic analysis of surfaces after cavitation led to the folowing conclusions:

- the surfaces affected by cavitation after 165 hours of immersion are approximately similar, within a narrow range, respectively 86.77% - 88.96%, there is no correlation between the holding time at 830°C and the surface of the cavitation attack;
- on the test probes, the existence of two distinct surfaces of cavitational attack is noticed, the general surface, of about 87.49% and a central surface, more defined as attack, of about 70%. The attack also generates intersecting ripples at different angles, a sign of a rather turbulent attack;
- for the specimens with the shortest holding time at 830°C, respectively 5 hours, the cavitational attack generates parallel ridges on extended areas with three different orientations: the attack area extends on about 86.77% of the specimen surface;
- for the test specimens treated at 830°C / 10 hours, it is also noted that for the test tube in delivery condition, the existence of two attack areas, respectively the general area of about 88.96% and the central area of about 68.85 %. At the same time, the arrangement of the front of the attack propagation at different angles is noticeable;

- for the specimens treated at 830°C / 20 hours, it is observed that the cavitation attack area is about 87.92%, identifying the central area less subject to cavitation attack;
- regardless of the heat treatment state applied at 830°C, or delivery state, all specimens are observed in the macrostructural analysis at magnification powers 56 times the appearance of similar cavities, with relatively evenly extended cavities in the field of observation. It is not possible to appreciate a differentiation neither in the size of the cavities, nor in their number;

➢ The analysis with the metallographic optical microscope could highlight the real value of the maximum penetration depth of the cavitational attack, observed by direct measurement. Regarding the cross-sectional appearance of the cavities observed on the INCONEL 718 specimens, in different structural states, it is noted that in all situations the cavities are fine, with a lacy appearance, with different depths, which could be correlated with the duration of heat treatment. aging at 830°C:

- The application of the aging treatment carried out at 830°C leads to the increase of the maximum depth of the cavity produced after the test 165 hours, the highest depth being registered for the specimens with aging at 830°C / 20 hours, respectively 6.5μm;
- Increasing the holding time to 830°C can cause an increase in the maximum cavity depth, but a rather insignificant increase, respectively from 5.1 μm to 6.5 μm, compared to the maximum depth of the control sample cavity, of 4.5 μm;
- In general, the values of the maximum penetration depths of the cavitation are particularly small, being located in the range of 4.5 ÷ 6.5 μm, compared to other classes of metallic materials;
- The analysis of the evolution of the values of the average erosion depths calculated MDEmax, expressed in μm compared to the values measured under a microscope of the cavity attack depth showed that the values have the same mode of evolution, there is some differentiation. The difference is that under the microscope the highest values were measured over the entire analyzed section, while the calculated values are average values, generated by a calculation module;

➢ Fractographic analysis under the scanning electron microscope of the specimens from the INCONEL 718 superalloy, in different structural states required for cavitation after 165 hours of immersion, highlighted the erosion of the surfaces allowed the formulation of the following aspects:

- At low magnification power at SEM, ie at macrostructural observation the surfaces are covered with fine parallel ridges, regardless of the state of the superalloy. However, in the sample in delivery, a disposition of the ridges at different angles is observed, compared to the other samples. On the other hand, it is found that as the maintenance time increases to 830°C, a slight intensification of the cavitation attack is observed, within reasonable limits;

- At higher magnification powers at SEM, ie at the microstructural observation the cavitational attack appears similar in all states of the superalloy, in the sense of the formation of small flow plates, with fine geometric cavities. As the maintenance time increases to 830°C, there is a numerical increase in the cavities, which tend to be arranged intergranularly (as noted at 830°C / 20 hours);
- It can be appreciated that at the longest holding time at 830°C, respectively 20 hours, the cavities are best formed, with intergranular appearance and average size of 3 ÷ 10μm;

➢ Conclusions regarding the cavitation behavior of X5CrNi18-10 stainless steel

- In the case of sensitization heat treatment, the cavitation resistance of X5CrNi18-10 steel depends on the technological parameters of the treatment, which influence the hardness and structure;
- Comparison with OH12NDL standard steel shows that, through the sensitizing heat treatment, the cavitation resistance of X5CrNi18-10 steel increases substantially, regardless of the condition, which is why this treatment, according to data from the Cavitation Laboratory of Politehnica Timișoara University, is recommended for parts working in developed cavity conditions;
- From the comparison with the volumetric heat treatment of tempering, due to the resistance to cavitation, lower, acquired by the heat sensitization treatment, it is recommended that for the parts strongly required in cavitation, such as those working in supercavitation mode) to use volumetric thermal hardening treatments for solution;

6.2 ORIGINAL CONTRIBUTIONS

➢ Carrying out a complete and complex structural analysis of the INCONEL 718 experimental specimens in different states, which allowed the evaluation of the dynamic recrystallization phenomenon and the temperature range at which it is present in the analyzed superalloy, as well as the evaluation of the cavitation resistance state.

➢ Carrying out its own experimental laboratory research on determining the behavior of hot plastic deformation in severe conditions, by testing the sonnet by free fall from different heights and at different temperatures, which allowed highlighting an interdependence between the grain size of the required specimen dynamic by free fall of the sonnet from h = 0.5 m, at different temperatures on the resistance to deformation, determined and calculated in laboratory experiments.

➢ Evaluating in an original manner the condition of the surfaces of the experimental deformability specimens by simultaneous analysis under a stereomicroscope and a scanning electron microscope.

- Carrying out an original complex analysis under the scanning electron microscope with the help of backscattered secondary electrons (SEM-EBSD) of the INCONEL 718 specimens required for compression by deformability tests in laboratory conditions which allowed obtaining results confirming stereomacrostructural observations, when determining the values of the distributions of the identified phases, namely γ, γ′, γ″ and MxCy, as well as the determination of the values of the average grain size, for both sampling directions, longitudinal and transverse;
- Carrying out a complex study on the behavior of cavitation erosion of the INCONEL 718 superalloy and assessing the intensity of its cavitation erosion phenomenon with an austenitic stainless steel type X5CrNi18-10, in different states of sensitization, as well as the experimental superalloy, all being compared with the cavitation erosion resistance of OH12NDL standard steel, stainless steel used in the manufacture of Kaplan turbine blades from the Iron Gates Hydroelectric Power Plant, appreciated as having good resistance to erosion produced by the vibrating cavity generated by the piezoceramic crystal device. The study allowed the formulation of original observations and contributions:
    - Regardless of the heat treatment state, both the INCONEL 718 superalloy and the X5CrNi18-10 austenitic stainless steel have clearly higher cavitation resistances than the OH12NDL standard stainless steel (increases from about 5 to 12 times, respectively 500% to 1200%);
    - by heat sensitization treatment at 650℃ / 30 minutes maintaining the cavitation resistance of austenitic stainless steel X5CrNi18-10 decreases by about 59% ;
    - by heat sensitization treatment at 650□C / 60 minutes maintaining the cavitation resistance of austenitic stainless steel X5CrNi18-10 decreases by about 56%;
    - by heat sensitization treatment at 750°C / 30 minutes maintaining the cavitation resistance of austenitic stainless steel X5CrNi18-10 decreases by about 121.6%;
    - by heat sensitization treatment at 750°C / 60 minutes maintaining the cavitation resistance of austenitic stainless steel X5CrNi18-10 decreases by about 36%;
    - The INCONEL 718 superalloy has the best cavitation resistance in all structural states, both compared to OH12NDL standard steel and X5CrNi18-10 stainless steel. The best cavitation resistance has the superalloy in the delivery state, with the cavitation resistance of over 1200% compared to the standard steel, and respectively 125% compared to the X5CrNi18-10 steel, in the delivery state; also, in each of the sensitization states, the INCONEL 718 superalloy has cavitation resistances between 700% and 600% compared to the standard steel, and respectively 233% and 165% compared to the cavitation resistance of the X5CrNi18-10 treated steel ;
- Innovative comparative presentation of cavitation attack depths, MDEmax (calculated) to absolute values δ (measured directly by light microscopy) in the form of a histogram.

6.3 DIRECTIONS AND PERSPECTIVES OF FUTURE RESEARCH

- During the research within the doctoral thesis, some aspects were identified, new or current, that can be considered or improved.
- INCONEL type superalloys are still sources of future experimental research on exploring their performance in the field of cavitation erosion, either under the same brand, INCONEL 718, but in other structural states of awareness than those used in these experiments, or other classes of INCONEL type superalloys.
- At the same time, the SEM-EBSD technique can be promoted on other structural classes of metallic materials strongly required for severe plastic deformation, in order to identify the definition of plastic deformation mechanisms.
- It may also be proposed to use the techniques specific to stereomicroscopy in assessing the condition of surfaces or plaster deformation, or the condition of surfaces required for cavitation erosion.

LIST OF PUBLISHED WORKS

1. GHIBAN B., Sohaciu M.G., Bordeasu I., Guragata C.M, Salcian L.C.- *HEAT TREATMENT AND CAVITATION BEHAVIOUR OF THE INCONEL 718 IN DIFFERENT HEAT TREATMENTS.* PROCEEDINGS OF RESEARCHFORA INTERNATIONAL CONFERENCE, Sydney, Australia, , 2020 (ISBN 978-93-89732-41-2, www.worldresearch.org): p. 4-9.

2. Ghiban, B; **Guragata, C. M**; Serban, N.; Raiciu, A. D.; Balkan, I.- HOT WORKING BEHAVIOUR OF INCONEL 718 SUPERALLOY , UNIVERSITY POLITEHNICA OF BUCHAREST SCIENTIFIC BULLETIN SERIES B-CHEMISTRY AND MATERIALS SCIENCE, Volume: 81, Issue: 4, Pages: 209-216, Published: 2019

3. Ghiban, B., **Guragata, C.M.,** Serban, N.- Particularities of forming behavior for INCONEL 718 superalloy, IOP Conference Series: Materials Science and EngineeringVolume 572, Issue 1, 2 August 2019, Article number 0120332019 International Conference on Innovative Research, ICIR EUROINVENT 2019; Palace of CultureIasi; Romania; 16 May 2019 through 17 May 2019; Code 150123

4. Ghiban, B.; Elefterie, C. F.; **Guragata, C**.; Bran, D.- Requirements of Inconel 718 Alloy or Aeronautical Applications , 7TH INTERNATIONAL CONFERENCE ON STRUCTURAL ANALYSIS OF ADVANCED MATERIALS (ICSAAM 2017), Edited by:Jiga, G, Book Series: AIP Conference Proceedings, Volume: 1932, Article Number: 030016, DOI: 10.1063/1.5024166, Published: 2018, Proceedings Paper

5. Elefterie, C. F.; **Guragata, C**; Bran, D; Ghiban, B.- Aeronautical requirements for Inconel 718 alloy, INTERNATIONAL CONFERENCE ON INNOVATIVE RESEARCH - ICIR EUROINVENT 2017, Edited by:Sandu, AV; Abdullah, MMA; Vizureanu, P; Ghazali, CMR; Sandu, I, Book Series: IOP Conference Series-Materials Science and Engineering, Volume: 209, Article Number: 012060, DOI: 10.1088/1757-899X/209/1/012060, Published: 2017, Proceedings Paper

SELECTIVE BIBLIOGRAPHY

1. Reed, R.C., *The Superalloys: Fundamentals and Applications.* Cambridge: Cambridge University Press, 2008

3. Antoniac I.,Laptoiu D., Blajan A.I., Cotrut C. -Instrumental si dispozitive chirurgicale, Editura Printech, Bucuresti, 2011, ISBN 978-606-521-665-5.

63. Bordeașu I., *Eroziunea cavitaţională asupra materialelor utilizate în construcţia maşinilor hidraulice şi elicelor navale. Efecte de scară.* Teză de doctorat, Timişoara, 1997.

64. Bordeaşu I., P.M.O., Patrascoiu C-tin, Bălăsoiu V., *„An Analytical Model for the Cavitation Erosion Characteristic Curves".* Transaction of Mechanics, Timisoara, 2004. **49(63**: p. 253-258.

65. Bordeașu I., *Eroziunea cavitaţională a materialelor.* Editura Politehnica, Timişoara, 2006.

66. Bordeaşu I., P.M.O., Patrascoiu C-tin, Bălăsoiu V., *„An Analytical Model for the Cavitation Erosion Characteristic Curves".* Transaction of Mechanics, Timisoara, 2004. **49(63**: p. 253-258.

125. Bordeasu, I., et al., *Chemical and mechanical aspects of the cavitation phenomena.* Revista De Chimie, 2007. **58**(12): p. 1300-1304.

126. Bordeasu, I., et al., *THE DAMAGE OF AUSTENITE - FERRITE STAINLESS STEELS BY CAVITATION EROSION.* Annals of Daaam For 2008 & Proceedings of the 19th International Daaam Symposium, 2008: p. 147-148.

127. Ghiban, B., et al., *SOME ASPECTS OF CAVITATION DAMAGES IN AUSTENITIC STAINLESS STEELS.* Annals of Daaam For 2008 & Proceedings of the 19th International Daaam Symposium, 2008: p. 541-542.

128. Ghiban, B., et al., *THE CALCULUS OF THE THERMODYNAMIC ACTIVITY OF CARBON IN SILICON ALLOYED AUSTENITIC STAINLESS STEELS.* Annals of Daaam For 2008 & Proceedings of the 19th International Daaam Symposium, 2008: p. 543-544.

129. Bordeaşu I., G.B., Popoviciu M. O., Bălaşoiu V., Birău N., Karabenciov A., *„The damage of austenite-ferrite steels by cavitation erosion".* Annals of DAAAM for 2008 Proceedings of The 19th International DAAAM Symposium "Intelligent Manufacturing & Automation: Focus on New Generation of Intelligent Systems and Solutions", 2008: p. 0147-0148.

130. Balasoiu, V., et al., *UPON CAVITATION EFFECTS IN HYDRAULIC DRIVING DEVICES.* Metalurgia International, 2009. **14**: p. 48-51.

131. Bordeasu, I., et al., *CONTRIBUTIONS UPON THE CAVITATION EROSION OF TWO CAST IRONS USED IN MANUFACTURING CONTROL VALVES.* Metalurgia International, 2009. **14**(11): p. 5-7.

132. Bordeaşu I., P.M.O., Sălcianu L.C., Ghera C., Micu L.M., Bădărău R., Iosif A., Pirvulescu L.D., Podoleanu C.E., *„A new concept for stainless steels ranking upon the resistance to cavitation erosion".* International Conference on Applied Science, 2017. **163**.

133. Ghiban, B., et al., *STRUCTURAL FEATURES OF CAVITATION DAMAGES IN SOME STAINLESS STEELS.* Annals of Daaam For 2009 & Proceedings of the 20th International Daaam Symposium, 2009. **20**: p. 1561-1562.

134. Ghiban, N., et al., *Evaluation of Mechanical Properties by Stereo-and Scanning Electron Microscopy of Some Heat Curing Dental Resins.* Materiale Plastice, 2010. **47**(2): p. 240-243.